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Synthesis of DDT Labeled with Carbon-14 in the Tertiary Position

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A method has been developed for the synthesis of radioactive Dichlorodiphenyltrichloroethane (DDT) in good yield in which the tertiary carbon is labeled.

Fields *et al.* (*1*) have developed a satisfactory procedure for incorporating C^{14} in the benzene ring. This procedure presumably was employed in a previously reported labeling of DDT with C^{14} (*2*). However, the complete synthesis of DDT by this procedure would involve at least seven major steps, with the consequent tendency towards low over-all yields. In the authors' procedure, only four major steps are necessary. Schematically, they are as follows:

$$BaC^*O_3 \longrightarrow CH_3C^*OOEt \xrightarrow{LiAlH_4} CH_3C^*H_2OH$$

$$Cl_2 \rightarrow CCl_3C^*HO \longrightarrow DDT$$

(the second step is the condensation with chlorobenzene, Cl–C$_6$H$_5$)

The synthesis of carboxy-labeled ethyl acetate was carried out by special adaptations of the methods of Sakami *et al.* (*3*), Van Bruggen *et al.* (*4*), and Ropp (*5*). The ester was reduced to ethyl alcohol by a modification of the method of Cox and Turner (*6*). Chlorination was carried out in a specially designed apparatus, and the subsequent condensation of chloral with chlorobenzene followed the work of Mosher *et al.* (*7*).

Starting with 50 mM of barium carbonate containing 20 mc of activity, 15 g of crude DDT (42% yield based on EtOH) was obtained. Two crystallizations from ethyl alcohol yielded 6.11 g of *p,p'* DDT (17% yield) having a melting point of 107–107.5° C. The specific activity of the product was approximately ½ mc/g.

The residue is currently being treated to recover additional amounts of the *p,p'*-isomer as well as the *o,p*-isomer.

It is planned to submit a detailed report of this work elsewhere.

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